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Laboratory Procedure for the Gravimetric Determination of Carbonate Carbon in Solid Wastes

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LABORATORY PROCEDURE FOR THE GRAVIMETRIC DETERMINATION OF CARBONATE CARBON IN SOLID WASTES

A Division of Research and Development
Open-File Report (RS-03-68-17)

U.S. ENVIRONMENTAL PROTECTION AGENCY
Solid Waste Management Office

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written by
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DISCUSSION

Inorganic carbon measurements on solid waste samples, in conjunction with total carbon measurements[1], serve as a criteria for evaluating incinerator efficiency to dispose of organic carbon matter. On the other hand, high carbonate content in incinerator residue will cause hardness in leachate water.

The carbonate analysis is also necessary because the carbonate content affects the analysis of other constituents and the analytical interpretation of other methods. Such as, the precision and accuracy of the ash-volatile determination[2] (loss-on-ignition or L.O.I. at 600C) can vary with the degree of decomposition of carbonates present.[3] Our laboratory investigations showed that during the calorific determination, the degree of decomposition of calcium carbonate in incinerator residue samples depends upon the amount of combustion aid added to the sample.[4] The carbonate content of samples must be known in order to calculate their calorific values from ultimate analyses. The oxygen content of samples is normally determined by subtracting from the volatile portion of a sample the carbon, hydrogen, nitrogen and sulfur contents. The oxygen values may be in great error because of the oxygen combined with the carbonates and metal oxides, which resulted from the decomposition of some carbonates, in the residue left from the volatile analysis.[5]

An existing A.O.A.C. method[6] for determining carbonate carbon was found feasible for solid waste samples.[7] Such sample with carbonate carbon contents from 0.05 to 8.00% have been precisely and accurately analyzed.

Before the carbonate carbon content is determined, all solid waste samples must be dried, ground to less than 2 mm and thoroughly mixed. Employing between 1 to 5 grams of sample in each determination produces satisfactory precise and accurate data.

Carbonate carbon is determined gravimetrically after: (1) reacting a weighed, dry, uniform sample with dilute hydrochloric acid inside a closed system and, (2) fixing the evolved gases in an absorption train (Figure 1). The procedure (Figure 2) is designed to measure the total carbonate carbon in dry solid waste samples.

APPARATUS

Requirements

1. Absorption bulb, Nesbitt, Pyrex; two or more (Fisher #7-517).*
2. Balance, analytical, 200 g capacity, 0.1 mg readability (Sartorius, 2400 Series).
3. Basket, test tube (Fisher #14-966D).
4. Beaker, Griffin low form, 250 ml.
5. Bottle, Gas Washing, Pyrex, with fritted cylinder, coarse porosity, 250 ml. capacity; two.
6. Bottle, narrow mouth, standard taper, 2000 ml.
7. Burner, gas.

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*Product (or manufacturer) identification shown in this Method does not imply endorsement by the Environmental Protection Agency.

8. Clamp, utility, vinylized jaws, three-prong grip; five.
9. Condenser, Kimble Modern Liebig, 400 mm. jacket (Fisher #7-704 C).
10. Cylinder, graduated, 50 ml.
11. Cylinder, graduated, 100 ml.
12. Cylinder, graduated, 500 ml.
13. Drying bulb, unique type (absorption bulb may be used).
14. Erlenmeyer flask, wide mouth, 125 ml.
15. Erlenmeyer flask, wide mouth, 250 ml; four or more.
16. Flask, filtering, side-arm, 50 ml.
17. Flask, filtering, side-arm, 250 ml.
18. Funnel, separatory, 60 ml.
19. Gloves, lint-free.
20. Hot plate (Fisher #11-494 or equivalent)
21. Rod, glass, about 6 inches long.
22. Rotameter or Flowrator Meter, size 02, sapphire float, hose connections, tri-float tube #02F-1/8-16-5 (Fischer and Porter #10A-1017 or equivalent).
23. Ring, support, with clamp, 4-inch outside diameter.
24. Stopcock, hard rubber (Fisher #14-630) (optional).
25. Stopper, one-hole, size #0.
26. Stopper, one-hole, size #5; two.
27. Stopper, one-hole, size #6; three.
28. Stopper, two-hole, size #6; two.
29. Stopper, two-hole, size #9.
30. Support stand, rectangular base, 24-inch rod; two.

31. Support stand, rectangular base, 36-inch rod.
32. Timer (Fisher #6-662).
33. Tube, glass, about one-inch diameter, about six-inches long.
34. Tubing, bubble, Argyle universal, plain, lumen, 3/16-inch bore (Aloe Scientific #AR-500).
35. Tubing, glass, 3/16-inch bore, about two feet.
36. Tubing, rubber, red, thin wall, 3/16-inch bore (Fisher #14-166).
37. Tubing, Tygon, 1/4-inch I.D., 3/8-inch O.D., 1/16-inch wall.
38. Tubing, vacuum-pressure, 3/8-inch I.D.
39. Vacuum source and regulator valve or clamp.
40. Wire square, asbestos center, 5-inch square.
41. Alkalimeter, Knoor, carbon dioxide apparatus (Fisher #1-198).
42. (Corequisite item no. 41) flask, Erlenmeyer form, with standard taper joint-24/40, Pyrex, 250 ml. (Fisher #10-047C).

NOTE: If items no. 41 and 42 are purchased then items no. 9, 15, 18, 25, two of 27, 29, and 33 need not be purchased.

Preparation

Assembling the train. The components of the apparatus are assembled in the following sequence:

a. Glass tube: Activated alumina is placed in the lower fourth and ascarite, topped with activated alumina, in the upper part. A small layer of glass wool is inserted beneath the lower activated alumina and above the upper activated alumina. The glass tube is closed at each end with one-hole, size #6 stopper. About one inch of glass tubing protrudes out from the hole in the stopper. The room air flow into the tube is through the botton inlet. The glass tube is attached to a 36-inch-rod support stand with a utility clamp.

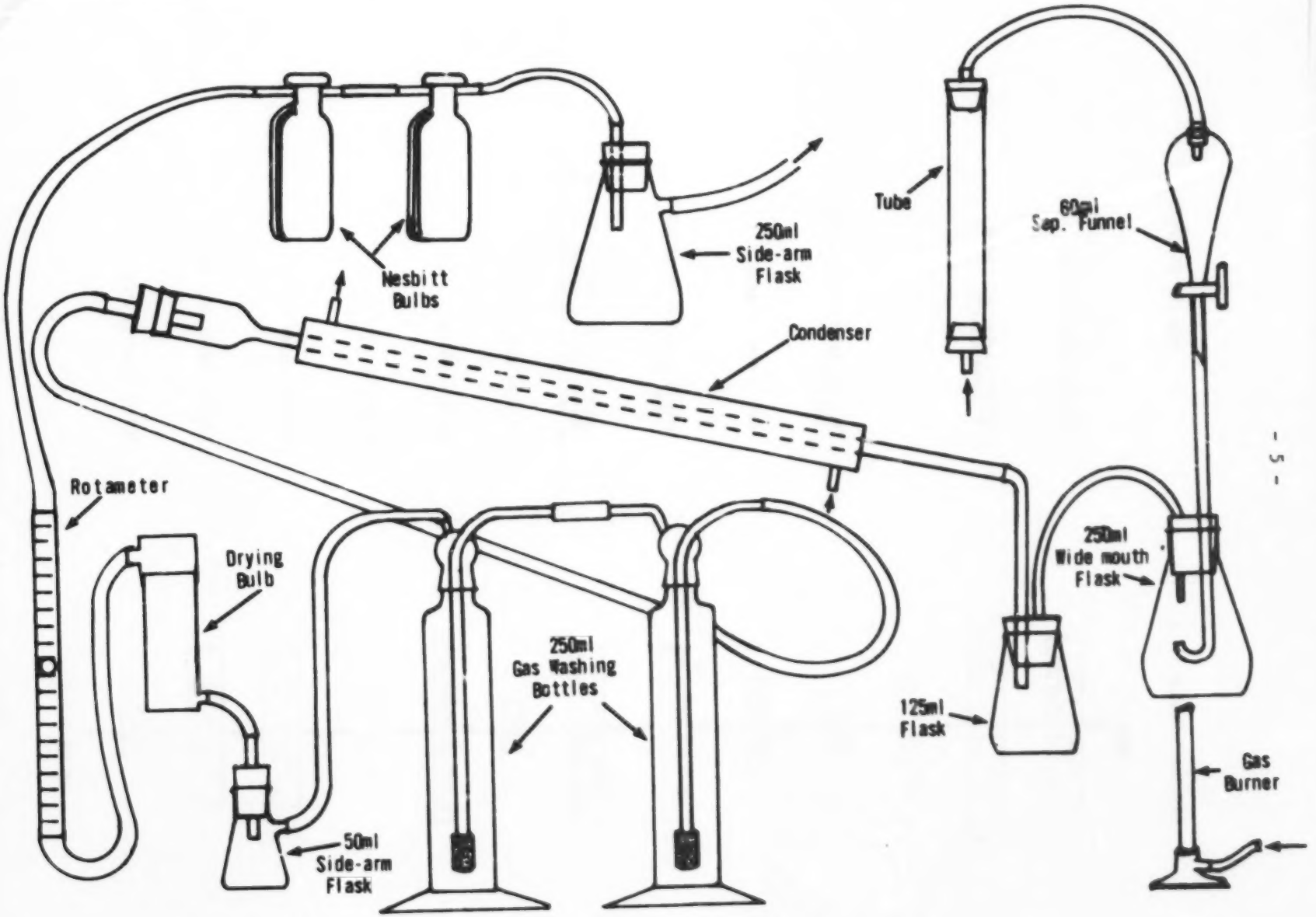

FIGURE 1. General schematic drawing of carbonate-carbon train.

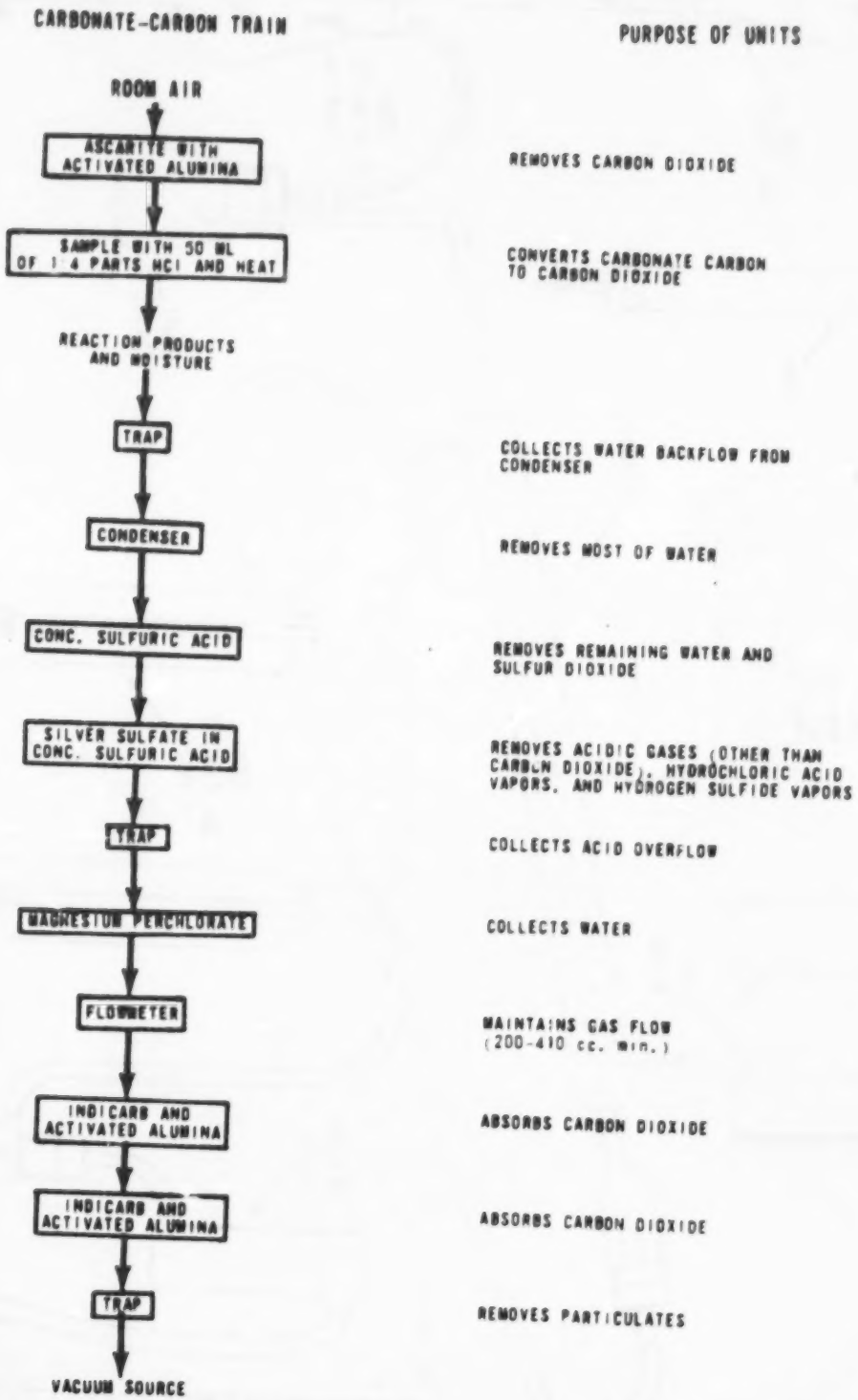

FIGURE 2 General outline of carbonate-carbon train.

b. Separatory funnel, 60 ml., with one-hole, size #0 stopper; attached to 36-inch-rod support stand with utility clamp.

c. Erlenmeyer flask, 250 ml., with two-hole, size #9 stopper and glass tubing through stopper holes; one glass tubing is bent upwards at end just enough to keep it from hitting the botton of flask; rest on wire square on ring support which is attached to 36-inch-rod support stand.

d. Gas burner sets under erlenmeyer flask (item c).

e. Erlenmeyer flask, 125 ml., with two-hole, size #6 stopper; glass tubing through stopper; (serves as a liquid trap). Note: If alkalimeter is employed this flask is after condenser of alkalimeter.

f. Condenser, 400 mm jacket, with one-hole, size #5 stopper at exit end; glass tubing through stopper; slanted at about 30° angle, with exit end at top; attached to 24-inch-rod support stand with utility clamp; Tygon tubing to water source and drain, with water entering lower part.

g. Alkalimeter (optional) replaces all items above except d and e.

h. Gas-washing bottle, containing 100 ml. concentrated sulfuric acid.

i. Stopcock, hard rubber (optional, helps prevent backflow of solutions).

j. Gas-washing bottle, containing 100 ml. silver sulfate solution.

k. Filtering flask, 50 ml., with one-hole, size #5 stopper; glass tubing through stopper; (serves as a liquid trap).

l. Drying bulb, containing magnesium perchlorate; small layer of glass wool beneath and above the magnesium perchlorate.

m. Rotameter, with the sapphire float between the 4.6 and 6.8 levels affording a flow between 200 and 410 cc/min.

n. Two Nesbitt bulbs (for carbon dioxide absorption), assembled in series (may be put in test tube basket); a 1/2-inch layer of glass wool is placed in the bottom of the bulb. Indicarb® is then added to a point, 3/4 inch from the shoulder of the bulb. The Indicarb® is covered by a 1/4-inch layer of activated alumina. Introduce sufficient glass wool to reach the neck area and cotton in hollow stopper. (Note: For highly reactive materials or materials with high carbonate carbon content more than two Nesbitt bulbs may be necessary.)

o. Filtering flask, 250 ml., with one-hole, size # 6 stopper; glass tubing through stopper; (serves as a particulate trap); connected to vacuum source and regulator with vacuum-pressure tubing.

p. Argyle-bubble tubing, used between glass tube, funnel, 250 ml.-erlenmeyer flask, 125 ml.-erlenmeyer flask, condenser, gas-washing bottles, 50 ml.-filtering flask, drying bulb, and rotameter.

q. Rubber tubing used between rotameter, Nesbitt bulbs, and 250 ml.-filtering flask.

r. Kel-F # 90 stopcock grease is used for all ground glass connections.

Conditioning the train. Unlike most trains, the carbonate carbon train requires very little conditioning. If the train has remained idle overnight, the analyst conditions the train by allowing room air to flow through the system at 200 to 410 cc/min. for about five minutes (see procedure).

REAGENTS

Chemical Requirements

The following chemicals are ACS Reagent grade:

1. Activated alumina, indicating, 8-14 mesh (Fisher #A-545).
2. Ascarite (sodium hydroxide in asbestos) 8-20 mesh.
3. Glass wool.
4. Sulfuric acid, concentrated.
5. Silver sulfate.
6. Magnesium perchlorate.
7. Indicarb®, 6-10 mesh (Fisher #1-181), (may be substituted with Ascarite).
8. Stopcock grease, Kel-F #90 (Sargent #S-77346).
9. Hydrochloric acid, concentrated.
10. Calcium carbonate.

Preparation of Solutions

1. Silver sulfate solution: Dissolve about 20.0 g Ag_2SO_4 in 100 ml (use graduate) of concentrated H_2SO_4 by putting both in a 250 ml beaker and heating on a hot plate, use glass rod to break up and mix Ag_2SO_4; note: Ag_2SO_4 decomposes at 652 C. Allow solution to cool to near room temperature before putting in gas-washing bottle. Final volume should be about 100 ml.
2. Dilute hydrochloric acid solution (1:4): Using a 500 ml graduated cylinder put 1600 ml of distilled water into a 2000 ml bottle. Add 400 ml of concentrated HCl and mix solutions by shaking and inverting the bottle.

Note: Prepare new solutions whenever used solutions foam excessively or analyses of standard plus impurities indicates depletion of silver sulfate effectiveness.

SAFETY PRECAUTIONS

Follow general laboratory safety rules. This method has no pronounced safety hazards. However, care must be taken when handling the concentrated acids.

SAMPLE PREPARATION

The details of sample preparation procedures which describe the drying and grinding techniques are not discussed in this laboratory procedural report. In general, raw refuse, incinerator residue (organics or combustibles), and compost are dried at 70 C to a constant weight and ground to a particle size of less than 1 to 2 mm. Incinerator residue (fines or noncombustible) and fly ash samples are dried at 105 C to a constant weight and pulverized to pass through a #60 (0.25 cm) sieve. Most of the metals, glass, and ceramics are removed before the samples are ground or pulverized. All samples must be well mixed before aliquots are removed for analysis.

PROCEDURE

Blanks

Increase in weight of the absorption bulbs is due to 1) sample-acid reaction, 2) sample flask and/or acid

contamination, 3) atmospheric contamination during sample container's connection to train, and 4) incomplete removal of carbon dioxide from the room air. The blank analyses determine the affects of all the above factors, except sample-acid reaction, upon the weights of absorption bulbs. These blank analyses are conducted as the regular sample analyses except no sample is used.

The analyst is advised to perform triplicate tests and repeat the blank analyses with each new batch of dilute hydrochloric acid solution.

Standard

The analyst should periodically check the carbonate carbon train by analyzing a standard, commonly calcium carbonate (previously dried at 105 C for one hour). The procedure for analyzing a standard is the same as described for sample analyses. However, analyze only 0.2 to 1.0 grams of calcium carbonate since this standard reacts quickly and easily with acid. A larger than 1.0 gram sample of calcium carbonate reacts too violently and causes gas to escape through the separatory funnel and into the room.

Samples

The following procedure applies to all solid waste materials, also to blanks and standards with the previously mentioned changes.

The analyst is advised to use 1.0 to 5.0 gram portions of samples for analysis. Sample weights up to ten grams may be used, if non-uniformity of sample warrants. Since the density of raw refuse, compost, and incinerator residue (organics or combustibles) samples

is low, the analyst need only weigh 1.0 to 3.0 gram portions of such samples.

Procedure	Comments
1. Transfer, at least, one to five (or one to three) grams of a solid waste sample into each of two 250 ml erlenmeyer flask. Determine and record the weight of each sample portion to the nearest 0.0001 g.	1. a) Duplicate determinations are sufficient for all solid waste samples. (See Method Evaluation) b) The type erlenmeyer flask depends upon whether or not the Knoor alkalimeter is employed.
2. With water going through condenser and stopcock of separatory funnel open, allow room air to flow through the carbonate carbon train at 200 to 410 cc/min. (If stopcock employed between gas-washing bottles, it must be open.)	2. a) The sapphire float in the rotameter should be between 4.6 and 6.8 levels. b) If conditioning of the train is needed, the air flow is maintained for about five minutes. c) An empty 250 ml erlenmeyer flask is used whenever a sample is not being analyzed.

3. Remove the absorption bulbs from the train and close each bulb to the atmosphere. Note: To prevent room air from entering the bulbs, disconnect bulb nearest vacuum source first.	3. A test tube basket is handy for carrying absorption bulbs. Bulbs may be left in basket while connected to the train.
4. Determine and record weight of each absorption bulb. These weights represent the initial weights of the absorption bulbs.	4. a) The bulbs should be near room temperature before being weighed. The reaction of CO_2 with the Indicarb® produces heat. b) Before weighing, each bulb is momentarily vented to the atmosphere and wiped clean with a lint-free cloth or glove. c) Use an analytical balance with a 200 gram capacity and 0.1 mg readability.
5. After opening the absorption bulbs to permit gas flow, quickly return the bulbs to the train assembly.	5. Start with the bulb furthest from the vacuum source. The connection of the bulbs to the vacuum source should be performed last.

6. After the rotameter indicates 200 to 410 cc/min. of gas is flowing through the train, remove the empty 250 ml erlenmeyer flask and connect a similar flask containing the weighed sample to be analyzed.	6. The sapphire float in the rotameter should be between 4.6 and 6.8 levels.
7. Close the stopcock of the separatory funnel.	7. This stopcock cannot be closed for several minutes.
8. With separatory funnel stopper removed add 50 ml (using 50 ml graduated cylinder) of dilute HCl solution (1:4).	8. If a Knorr apparatus is used, the drying tube atop the funnel must be removed to add the acid solution.
9. Replace separatory funnel stopper.	
10. By turning stopcock, slowly add dilute acid to 250 ml erlenmeyer flask.	10. If the sample is calcium carbonate or very high in carbonate addition of acid to sample is very-very slow.
11. Immediately after all acid enters flask, close stopcock of funnel.	11. If not closed immediately, possible loss of CO_2.

12. Apply heat until sample and acid solution starts to boil.	12. Be sure all the acid solution comes into contact with the sample. (Flask may need swirling.)
13. With heat removed, slowly open stopcock of funnel, keeping rotameter float between 4.6 and 6.8 levels.	13. The flow rate should be maintained between 200 and 410 cc/min.
14. With the stopcock completely open and the flow rate adjusted, set a timer for 20 minutes.	14. A 20-minute flush time is sufficient for every type sample.
15. After the 20-minute flush time, stop the flow by removing the rubber tubing from the last absorption bulb. Note: If a stopcock is used between gas-washing bottles it is closed.	15. If another connection is separated first, then room air will enter absorption bulbs.
16. Remove all the absorption bulbs from the train and close each to the atmosphere.	16. If necessary, the train may remain idle for several hours.

17. As before, determine and record the weight of each absorption bulb.

17. This weight represents the final weight of each absorption bulb and is used as the initial weight of each bulb for the next sample.

18. If another sample is to be analyzed, repeat the procedure starting with step #5. (If stopcock employed between gas-washing bottles it must be open.)

19. If the train is not to be used for the remainder of the day, turn "off" water to condenser, close vacuum source, shut "off" gas, and close all stopcocks.

CALCULATIONS

Standards

Employ the following formula to calculate the theoretical concentration of carbon in a standard sample:

$$\% C = \frac{(N)(F)(100)}{(S)(P)}$$

Where % = The percent by weight

C = The element carbon

N = The number of atoms of the element in a molecule of the standard

F = A factor, derived by dividing the gram-atomic weight of the element by the gram-molecular weight of the standard

S = The weight of the total sample

P = The decimal fraction representing the concentration of the standard compound in the total analyzed sample (Note: this decimal fraction is the only fraction containing the component being analyzed for)

Example:

Pure Calcium Carbonate $Ca\ CO_3$

$$\% C = \frac{(1)\ [\frac{12.01}{100.09}]\ (100)}{(1.0000)\ (1.00)} = 12.01$$

When the impurities listed on the bottle are considered, the calculated percent of carbon in ACS grade calcium carbonate is still 12.01. (ACS grade about 99.956% pure)

Samples

Employ the following formula to calculate the concentration of carbon in a solid wastes sample:

$$\% C = \frac{(A-B)\ (X)\ (100)}{(S)}$$

Where % = The percent by weight

C = The element carbon

A = The sum total increase in the weight of the absorbing bulbs as determined in the unknown analyses

B = The sum total increase in the weight of the absorbing bulbs as determined in the blank analyses

X = A factor, derived by dividing the gram-atomic weight of carbon by the gram-molecular weight of carbon dioxide; i.e., (12.01)÷(44.01)=0.2729

S = The weight of the total sample

METHOD EVALUATION

The accuracy of this method was established by analyzing ACS grade calcium carbonate eight times. The average percent carbon found was 11.97. (The calculated percent carbon value is 12.01.) The standard deviation for these eight observations was 0.18. This method can analyze solid wastes materials, containing various forms of carbon or excessive amounts of interferences, to within 0.02 to 0.50 (depending upon type* of sample) actual percent of the established value. For the forms of carbon, sucrose and urea were selected to represent organic carbon. Graphite was employed as elemented form of carbon. Potassium fluoride, sodium sulfate, sodium chloride, and sodium nitrite were employed as the interfering materials. This method should not be employed to analyze samples which contain less than 0.01 percent carbonate carbon.

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* Types of solid wastes in this paper refer to only solid sample (domestic origin) as: raw refuse, incinerator fly ash, incinerator residue, and compost.

The precision (pooled standard deviation) of this method was determined by analyzing, in triplicate, a number of solid waste samples of various types. The pooled standard deviation of the observations for each type of solid wastes was calculated using an Olivetti Underwood Programma 101. The calculations revealed that, in the analyses of each type of wastes, the duplicate and triplicate determinations were about equally precise (Table 1). To ensure good precision the particle size of the samples must be less than 2mm or pass through a #60 sieve, then thoroughly mixed before analyzing.

With this method the analyst normally uses a one to five-gram sample, but he is not restricted to this amount. Because of the difficulties in preparing a very uniform sample, sample weights below one gram have been found inadequate when analyzing solid waste materials. But samples up to ten grams have been analyzed with no difficulty. The extra sample weights, however, add little to the precision of this method.

The performance of this method requires only periodic attention by the analyst. At best, eight samples a day can be analyzed in duplicate (about thirty minutes per determination), leaving the analyst with approximately two hours of free time per day.

TABLE 1

STANDARD DEVIATION[a] OF THE CARBON (CARBONATE) DETERMINATION ON CALCIUM CARBONATE AND SOLID WASTES

Type of Sample	Number of Samples	Carbon Duplicates	Carbon Triplicates	Percent Carbon Range
Calcium Carbonate[b]	3	-	0.18	-
Residue (Fines)[c]	12	0.01	0.02	0.05 to 0.69
Residue (Organics)[d]	4	0.18	0.15	0.59 to 8.00
Fly Ash	6	0.13	0.15	0.70 to 1.39
Raw Refuse	13	0.08	0.07	0.06 to 0.96

[a] A variance estimate can be calculated from the duplicate (or triplicate) set of observations for each sample. The pooled variance is essentially an average of all such estimates for samples of a given type. It is assumed that a single underlying variance exists for all samples of a given type. The pooled variance is then the best estimate of this underlying variance. The pooled standard deviation is the square root of the pooled variance and is used to estimate the underlying standard deviation.

[b] American Chemical Society grade.

[c] "Fines" are materials remaining after most of the readily combustible substances have been removed by manual sorting. The sorting was performed at the incinerator cites and a 1/2 inch sieve was employed to assist in the separation.

[d] "Organics" or "Combustibles" are mostly the readily combustible materials. Unlike "Fines" these materials are usually retained on a 1/2 inch sieve.

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The author gratefully acknowledges the contribution of Israel Cohen, Research Services Laboratory, Solid Waste Management Office, who prepared many of the samples used in developing this method.

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